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Procedia Materials Science 5 (2014) 444 – 453
Procedia Materials Science
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International Conference on Advances in Manufacturing and Materials Engineering, AMME 2014

# Effect of T6 heat treatment in the microstructure and mechanical properties of A356 reinforced with nano $Al_2O_3$ particles by combination effect of stir and squeeze casting

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**Abstract**

Hypoeutectic aluminum silicon alloys as (A356) exhibit several specific and interesting properties that qualify them to be used in many automotive and aeronautical applications, aluminum matrix composites have good physical and mechanical properties, when reinforced with nano sized $Al_2O_3$ particle, they have high tensile, compressive and fatigue strengths along with superior wear resistance and thermal stability. In this work, the number of cast samples of A356 alloy was prepared by combination effect of stir and squeeze casting, specially designed and built furnace unit allowing for the addition of $Al_2O_3$ nano-particles (30 nm). The percentage inclusions were varied from 0.5 to 1.5wt% into the molten Al-Si alloy in the liquid state with stirring at 400 rpm and squeeze casting at 750$^0$C and pressure of 600 MPa. The microstructural features and the mechanical properties of the as cast and T6 heat treated samples were investigated. The results obtained in the work showed enhance in the mechanical properties of the $Al_2O_3$ nano-dispersed alloys. Optical microscopic examination revealed uniform distribution of nano particles. Measurements of hardness, compressive strength and double shear strength showed an increase with increase in percentage addition of these nano particles.


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*Keywords:* A356 - $Al_2O_3$ nano composite, Stir and Squeeze casting, Microstructure, Hardness, Compressive strength, Double shear strength.

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Selection and peer-review under responsibility of Organizing Committee of AMME 2014
doi:10.1016/j.mspro.2014.07.287

## 1. Introduction

Among different aluminum alloys, A356 alloys are widely used commercially in industries like automobile and other applications because of its low density, electrical conductivity and corrosion resistance. The present needs for light weight materials with high strength and high stiffness have attracted much interest in the development of processes for developing metal-matrix composites. Hence, an attempt to go for A356 alloy reinforced with micro and nano $Al_2O_3$ particles by Squeeze casting with stirring is made here. As stir cast components are reported to have superior mechanical properties, fine microstructure and minimal porosity [Ghomashchi and Vikhrov, 2000]. EL-Mahallawi et.al influence of $Al_2O_3$ nano-dispersions on microstructure features and mechanical properties of cast and T6 heat-treated Al Si hypoeutectic Alloys [Mahallawi et al., 2012].Their results obtained enhancement in the mechanical strength of the $Al_2O_3$ nano-dispersed alloys .Nano-particles could be successfully incorporated into semi solid slurries of Al-Si hypoeutectic alloys by including mechanical stirring, through more work need to be done to achieve conditions for homogeneous dispersion. A study of published works showed that A356 alloy reinforced with micro and nano $Al_2O_3$ particles composite fabricated using compocasting [Sajjadi et al., 2012], the hardness of the composite increased with increasing particle weight percentage. D.Emadi et.al optimal heat treatment of A356.2 alloy[Emadi et al.,2003], their results showed the case can be made for the 155°C aging treatment based on dimensional changes and reproducibility in properties of the lower aging treatment temperature, this treatment when coupled with solutionizing 4 hours at 540°C , cold/warm water quench and 6-12 hours pre-aging optimal properties and reproducibility. Stir casting method was used to make the A356/$Al_2O_3$ micro and nano composites [Sajjadi et al. 2011] which revealed that the nano composites exhibited better properties in terms of compressive strength, hardness with reduced porosity. Peng Ji-hua et.al, effect of heat treatment on microstructure and tensile properties of A356 alloys [Peng Ji-hua et al., 2011], their results showed the solution at 535°C for 4hr and the solution at 550°C for 2hr can reach full spheroidization of Si particle, over saturation of Si and Mg in alpha (Al). The heat treatments of T6 and short time (ST) produce almost same microstructure of A356 alloy. Results on A356/MgO nano composites fabricated using stir casting method [Ansary Yar 2009] showed that the composite containing 1.5 vol % MgO particles at 850 $^0$C have homogenous micro structure as well as improved mechanical properties such as hardness and compressive strength. The bending strength and elastic moduli of A356 reinforced with alumina particles fabricated by squeeze casting also showed improvements [Shuangie chu and Renjie wuu, 1999].

Ezatpour at.al [2011] microstructure and mechanical properties so Al- $Al_2O_3$ micro and nano composites fabricated by a novel stir casting route,the result showed that heat treated micro/nano $Al_2O_3$ particles, injection of particles and stirring system were improved the wettability and distribution of the nano particles within the aluminum melt. Also, it was revealed that the amount of hardness and porosity increased as weight percentage of nano $Al_2O_3$ particles increased.Report on rheocasting combined with squeeze casting techniques to make A356/ $Al_2O_3$ nano composites [El-sayed yousef et al., 2011] showed that the increase in the content of $Al_2O_3$ nano particles on A356 aluminum alloy increased both tensile and yield strength of composite with finer size of nano particles. Results on fabrication and studying the mechanical properties of A356 alloy reinforced with $Al_2O_3$ -10% vol.$ZrO_2$ nano particles through stir casting [Mohsenhagizamani & Hamydrez Baharvandi 2011], revealed that with increasing the reinforcement content density decreased while yield, ultimate tensile strength and compressive strength increased. Also hardness increased by increasing the reinforcement content up to 1wt% $Al_2O_3$ -10% vol.$ZrO_2$ , but it decreased in the samples containing higher amounts of reinforcement. Stir and compo casting technique have been used to analyze microstructure and mechanical properties of $Al_2O_3$/A356 nano composite [Sajjadi et al., 2012]. It was identified that significant improvement in strength and hardness was shown with 3 wt. % nano $Al_2O_3$ for compocasting and with 2 %wt. nano particles. The most important challenge during fabrication of MMCs by liquid phase processes are uniform mixing of reinforcement in the matrix without sinks and floats, wettability of ceramic particles in the base metal with less porosity and higher density. The aim of the present investigation is to study the microstructure and mechanical properties of the cast and T6 heat-treated A356 Al-Si alloy reinforced with $Al_2O_3$ nano particles. To overcome these challenges, by adding the nano particles by stir with squeeze casting procedure were adopted. literature discusses the mechanical properties in terms of microstructure, hardness, tensile and compressive strength. The aim of the present investigation is to study the microstructure and mechanical properties of the as cast and T6 heat-treated A356 alloy reinforced with $Al_2O_3$ nano particles. The present work elaborate composites behavior during double shear test.

## 2. Experimental Procedure

### *2.1 Materials and Manufacturing Process*

The matrix alloy used for this study is A356 alloy which has chemical composition in weight percentage as in Table 1.$Al_2O_3$ particles of 30nm size were added as the reinforcements in the metal matrix composites.

Table 1. Chemical composition of A356 alloy

| Chemical compositions | Cu | Si | Mg | Mn | Fe | Ti | Ni | Zn | Tin |
|---|---|---|---|---|---|---|---|---|---|
| Wt % | 0.099 | 6.65 | 0.55 | 0.06 | 0.32 | 0.061 | 0.04 | 0.004 | <0.001 |

The chemical composition analysis was carried out as per ASTM E1251-07 OES standards. The A356 alloy billets were heated in electric furnace up to 750 $^0$C. After complete melting of aluminum alloy, the stirrer was brought down into the molten metal and rotated at a constant speed of 400 rpm. $Al_2O_3$ nano particles were preheated at 900 $^0$C to improve the wettability in the molten base metal. After preheating, the particles were slowly injected in to the molten metal by taper pipe arrangement provided above the melting vessel of the furnace. The stirring action was continued for 10 min to improve uniform distribution of the $Al_2O_3$ nano particles. After melting, bottom pouring valve of the furnace was operated using automatic control to pour the molten metal into the die steel mold. At this moment, squeeze piston was simultaneously activated to squeeze the molten nano composite metal. After squeezing the molten metal, the die setup was cooled slowly by circulating the water around the die wall which enhances the mechanical properties of the casting by the cooling effect. The final solidified nano composite was taken out from the die. Fig.1, shows the final blank after squeeze casting with the diameter of 46 mm and length 260 mm.

Fig. 1. Final cast composite blank

### *2.2. Specimens preparation*

Different samples of the cast and T6 heat treatment were prepared for micro structural analysis, hardness, compression and double shear test. SiC emery papers of various grades up to 1200 grit size in wet condition were used for polishing. The fine polishing of specimens were carried out using alumina paste and diamond paste of 1 µm in size. As per ASTM E3 standard, the fine polished samples were etched using Keller's reagent (2.5% $HNO_3$ + 1.5% Hcl + 1% HF + 95% $H_2O$). The micro structural examination was carried using optical microscope with 200 X magnification. Hardness was measured on the as cast and T6 heat treated samples using Rockwell hardness tester. The tests were carried out by applying a minor load of 10 kg and a major load of 100 kg with a steel ball of radius 1.588 mm for 15 seconds. Four readings were taken in a Rockwell B scale, in each specimen and the average value was considered. Compression tests were conducted on nano composite specimens of the as cast and T6 heat treatment samples using INSTRON compression testing machine according to the ASTM E9-X standards. The specimen had height of 15 mm and diameter 10mm, (aspect ratio of 1.5). Double shear tests were conducted on the as cast and T6 heat treatment samples using 200 kN capacities universal testing machine (UTM) with the help of double shear chuck arrangement. Sample sizes were 10 mm in diameter and 100 mm in length as per ASTM B769-11 standards.

## 3. Results and Discussions

*3.1 Microstructure observations of as cast samples*

Typical optical microscope images showing the microstructure of A356 and A356/ $Al_2O_3$ nano composite are as in Fig.2,

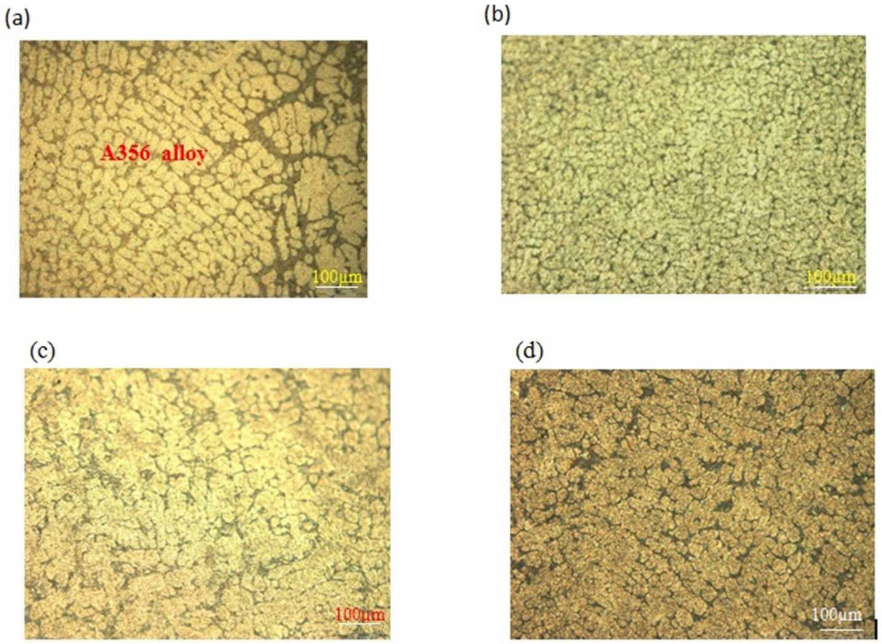


Fig. 2. Microstructure image of the base alloy and composites with 200X magnification (a) A356 Al alloy, (b) Al+0.5% $Al_20_3$, (c) Al+1 % $Al_20_3$, and (d) Al+1.5 % $Al_20_3$

Fig .2, (a-d) shows the A356 alloys and composites having 0.5, 1 and 1.5 wt% of $Al_2O_3$ reinforcements.The microstructures revealed that the composites have dense surface without micro level cavities due to squeezing the molten metal and uniform distribution of $Al_2O_3$ nano particles on the aluminum grains. Composites with lesser wt% of $Al_2O_3$ showed better and uniform distribution of nano particles. The grain size reduces because of the particles act as nucleation sites; the particle size reduces due to good wettability of the particles in the melt by preheated nano particles. In case of composite with 1.5 wt% nano particles, the formation of clustering effect of particles in different regions could be observed.

*3.2 Microstructure observations of T6 heat treated samples.*

Fig .3, (a-d) shows the A356 alloys and composites having 0.5, 1 and 1.5 wt% of $Al_2O_3$ reinforcements. The following optical microscope images shows the microstructure of A356 and A356/ $Al_2O_3$ nano composites in T6 heat treated condition with 200 X magnification. The hypo-eutectic based materials were subjected to T6 heat treatment condition (solution treating at 460 $^0$C for 6 hours and ageing at 190 $^0$C for 4hours). The microstructures revealed that the composites have dense surface without micro level cavities due to squeezing the molten metal and uniform distribution of $Al_2O_3$ nano particles on the aluminum grains. Composites with lesser wt% of $Al_2O_3$ showed better and uniform distribution of nano particles. The coarse grain structure of heat treated specimen is due to the recrystallisation and grain growth is observed by the nano particles. The white region in the microstructure is the matrix and the grey region is the eutectic silicon. The large size of the eutectic silicon phase will degrade the strength of the casting. The silicon phase is brittle, so the crack will first generate in silicon and propagate rapidly through the $Al_2O_3$ nano particles, the matrix leading to failure.

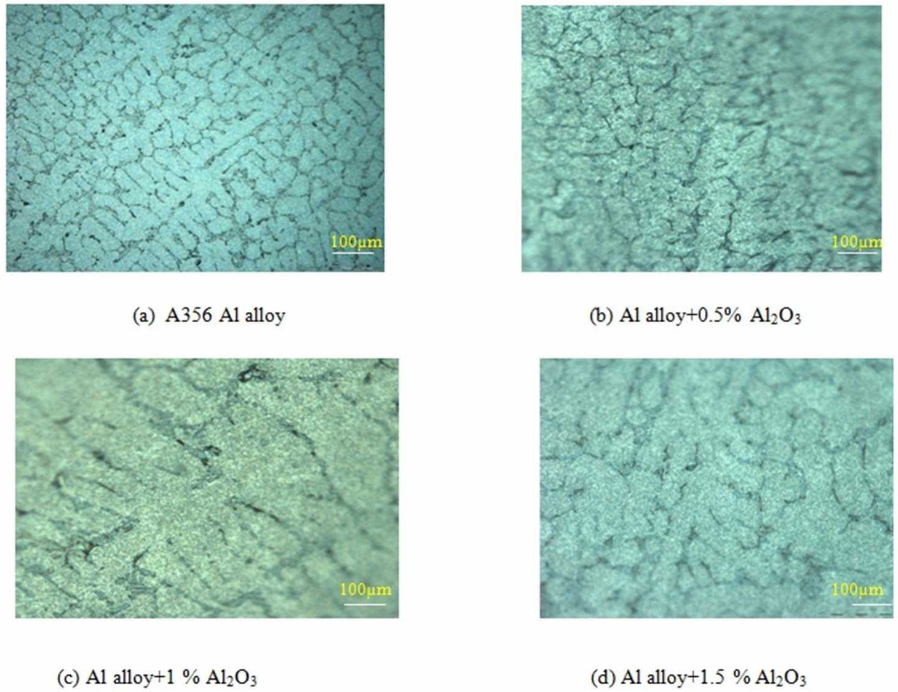


Fig .3. Microstructure image of T6 heat treated samples of the base alloy and composites

*3.3 Hardness*

Fig .4, Shows the variation of Rockwell hardness of the nano composite with increase in volume fraction of $Al_2O_3$ nano particles in the A356 matrix. It was observed that addition of nano composites of 0.5, 1 and 1.5 wt% resulted in higher hardness than the A356 monolithic alloy, because of uniform distribution of reinforcement and resistance to deformation from the ceramic phases. By adding 0.5 %, 1.0 % and 1.5 % wt% of $Al_2O_3$ nana particles in the composite caused the percentage increase in hardness values 34.8, 42.2 and 24 respectively. The composite containing of 1 wt% $Al_2O_3$ showed maximum hardness of 70 HRB. Beyond 1 % of $Al_2O_3$ nano particles addition (1.5 wt%) the tendency was decreased from 70 HRB to 62 HRB due to clustering effect of nano particles within the aluminum alloys.

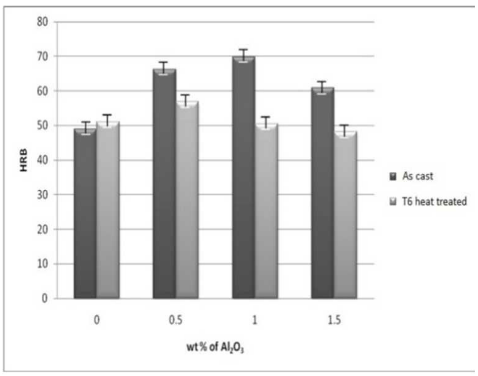


Fig.4, Variation of hardness

*3.4 T6 heat treated samples hardness*

Fig .4, shows the variation of Rockwell hardness of the nano composite with increase in volume fraction of $Al_2O_3$ nano particles in the A356 matrix in heat treated condition. It was observed that addition of nano composites of wt% 0.5 and 1 resulted in higher hardness because of uniform distribution of reinforcement and resistance to deformation from the ceramic phases. The composite containing of 0.5 wt% $Al_2O_3$ showed maximum hardness of 58 HRB. Beyond 0.5 wt. % of $Al_2O_3$ nano particles addition (1wt %) the tendency was a decrease in the value from 58 HRB to 50HRB because in T6 heat treatment more addition of $Al_2O_3$ nano particle (1.5 wt %) not dissolve in solid solution at 400 $^0$C -540 $^0$C. By adding 0.5 wt% $Al_2O_3$, the percentage increase in hardness value in T6 heat treatment is 11.7.

*3.5 Compressive test results for as cast samples*

The results of compressive strength of as cast and MMC samples are shown in Fig .5, The addition of nano particles to the A356 alloy showed increase in compressive strength initially and then a decrease. The grain refinement has taken place in the specimens with 0.5% $Al_2O_3$ is due to the fact that the particle act as nucleation sites and the pressure applied during solidification break up the agglomerates with reduce the porosity. Composite with 0.5 % $Al_2O_3$ showed highest value due to the higher uniform distribution of the nano particles compared to the other composites since the particle are distributed reasonably at this %. The average yield compressive stress (YCS) value of three samples in each materials and the corresponding standard deviation (σ) of the measurement are listed in Table 2. The results indicate that the effect is due to the change in property as observed by the lower standard deviation.

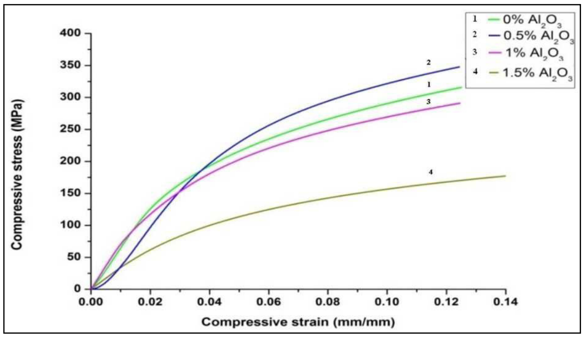


Fig. 5. Variation of compressive strength of as cast samples

Table 2. Average value of compressive stress (MPa) and standard deviation

| Materials | YCS (average) | Standard deviation |
|---|---|---|
| A356 alloy | 181 | 1.2 |
| Al+0.5% $Al_20_3$ | 215 | 3.1 |
| Al+1 % $Al_20_3$ | 148 | 3.7 |
| Al+1.5 % $Al_20_3$ | 111 | 2.6 |

*3.6 Compressive test results for T6 heat treated samples*

The results of compressive strength of the Al and heat treated (HT) MMC composites are as shown in Fig .6, The addition of nano particles to the A356 alloy showed the decrease in compressive strength initially and then an increase. The lower compressive strength at 0.5 % of $Al_2O_3$ nano particles is due to the less resistance offered by the hard ceramic particle against the applied load during test. The higher strength is due to better interface bonding characteristics of the matrix and the reinforcement. Lower compressive strength at higher (1.5 wt %) $Al_2O_3$ is due to more agglomeration and particles did not dissolve in the solid solution at 400 $^0$C to 540 $^0$C. Compressive with 1wt% $Al_2O_3$ showed higher value due to the higher uniform distribution of the nano particles compared to other composites. The average yield compressive stress (YCS) value of three samples in each materials and the corresponding standard deviation (σ) of the measurement are listed in Table 3. The results indicate the effect is due to the change in property as evidenced by the lower standard deviation.

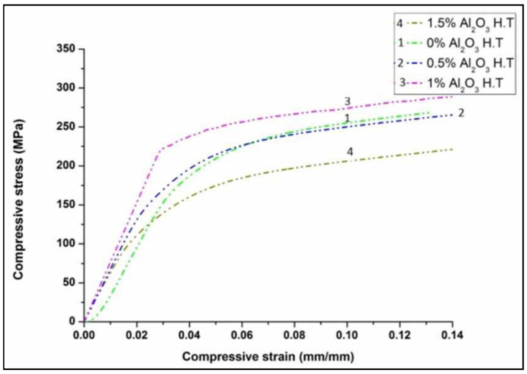


Fig. 6. Variation of compressive strength for T6 heat treated samples

Table 3. verage value of compressive stress (MPa) and standard deviation

| Materials | YCS (average) | Standard deviation |
|---|---|---|
| A356 alloy | 160 | 2.5 |
| Al+0.5% $Al_20_3$ | 140 | 2 |
| Al+1 % $Al_20_3$ | 220 | 4 |
| Al+1.5 % $Al_20_3$ | 110 | 2.4 |

*3.7 Double Shear test results for as cast samples*

Fig.7, shows the double shear strength of the A356/ $Al_2O_3$ nano composites with three different wt% of nano particles in as cast condition. The double shear strength increased with increase in addition of $Al_2O_3$ nano particles from 0 to 1wt% and after that it showed a decreased trend. The density of dislocation is very high in uniformly distributed samples due to high interfacial area, buy adding 1 wt% of $Al_2O_3$ nano particles; double shear strength is increased by 13.74%. The increase in double shear strength is due to more uniform distribution of nano ceramic particles in the aluminum alloy and resistance offered by the hard ceramic particle against the applied load during shearing. Above 1wt% of $Al_2O_3$ nano particle addition, the double shear strength started to decrease due to clustering effect of nano particles on the matrix alloy which weakens the strength of the composites.

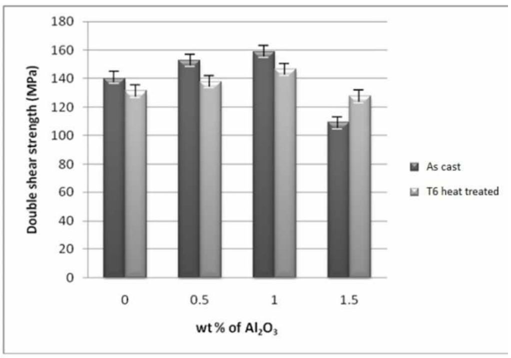


Fig. 7. Variation of double shear strength (as cast and T6 heat treated samples)

*3.8 Double shear test results for T6 heat treated samples*

Fig .7, shows the double shear strength of the T6 heat treated samples of A356/ $Al_2O_3$ nano composites with three different wt% of nano particles. The double shear strength increased with increase in addition of $Al_2O_3$ nano particles from 0 to 1wt% and after that it showed a decreasing trend. With T6 heat treatment, the double shear strength decreased due to the week interface bonding characteristics between the matrix and reinforcement. Above 1wt% of $Al_2O_3$ nano particle addition, the double shear strength started to increase due to the inability to dissolve in solid solution.

*3.9 Fracture surface of double shear test for as cast samples*

Figure.8 shows the SEM image of double shear fracture studies of the A356/Al2O3 nano composites with three different wt % of nano particles in as cast and T6 heat treated condition. By increasing the wt% of Al2O3 nano particles reinforcement, the fracture surface shows the presence of equi-axed shallow dimples indicates the low ductility of the materials, due to fracture of the reinforcing particles, partial de-bonding of particle-matrix interface and nucleation of voids, growth of the voids and initiation of cracks in the matrix.

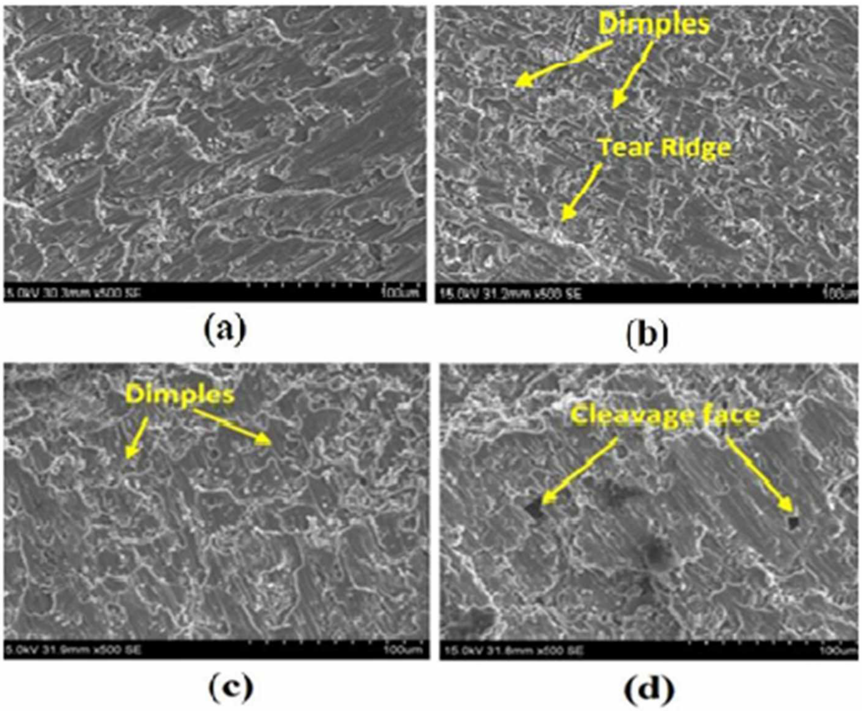


Fig 8. SEM Photographs of double shear fractured surfaces of as cast (a) A356 Al alloy, (b) Al+0.5% $Al_20_3$, (c) Al+1 % $Al_20_3$, and (d) Al+1.5 % $Al_20_3$

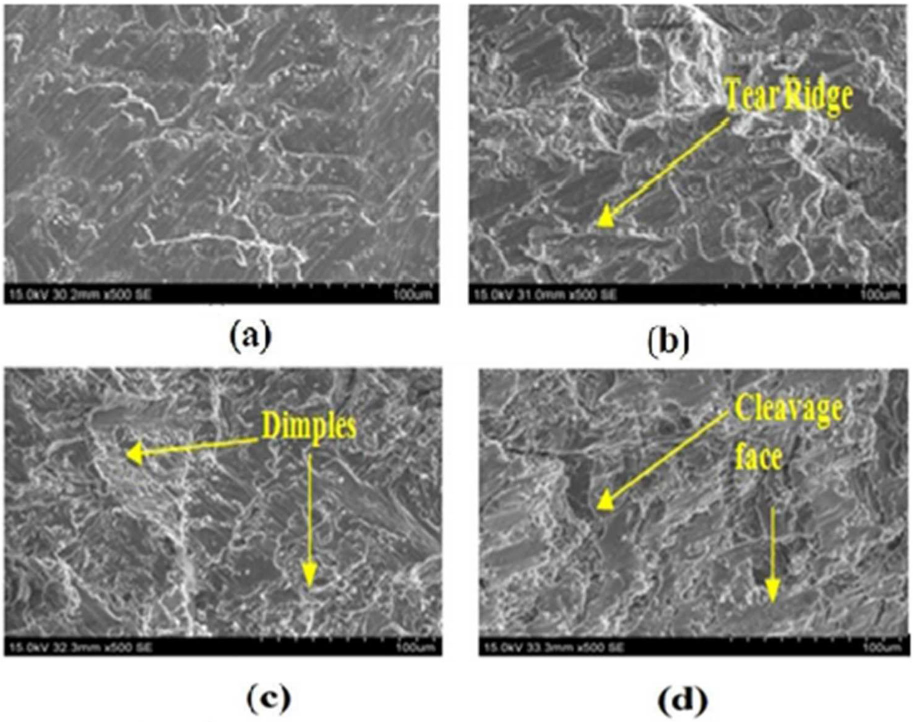


Fig 9. T6 heat treatment SEM Photographs of double shear fractured surfaces (a) A356 Al alloy, (b) Al+0.5% $Al_20_3$, (c) Al+1 % $Al_20_3$, and (d) Al+1.5 % $Al_20_3$

A356 shows pure inter-granular fracture. It has been observed that the plastic flow has been limited with increase the ceramic addition of Al2O3 nano particle, which resulted in brittle failure of the composites. For composite with 1.5 wt% Al2O3 nano particles create the cleavage face is clearly seen in the as cast condition in Figure 8. It shows inter-granular fracture. The mode of fracture is more brittle fracture than ductile fracture. More addition of nano particles, the agglomeration is formed inside the materials, therefore this agglomerated nano particles easily come out in the form of cleavage face from the materials during double shear test. With T6 heat treatment, the double shear strength decreased due to the week interface bonding characteristics between the matrix and reinforcement. Above 1wt% of $Al_2O_3$ nano particle addition, the double shear strength started to slightly increase due to the inability to dissolve in solid solution. Here reduced the cleavage face is clearly seen in the fracture surface image of T6 condition in figure 9.

## 4. Conclusions

Based on the results obtained in this work the following conclusions may be drawn:

1. $Al_2O_3$ nano-particles could be successfully incorporated into liquid metal of Al-Si hypoeutectic alloys by stirring, though more work need to done to achieve homogenous dispersion.
2. Introducing $Al_2O_3$ nano-particles to the Al-Si liquid metal cast alloys produce an increase in hardness with increase in the wt% of $Al_2O_3$ nano-particles. This increase is due to the existence of hard $Al_2O_3$ nano-particles which act as an obstacle to the motion of the dislocation is due to addition of 0.5, 1.0 and 1.5wt % $Al_2O_3$ nano-particle in the as cast state. The percentage of increase in the hardness values are 34.8, 42.2 and 24 respectively and produce a hardness value of 11.7 with T6 heat treatment condition.
3. The compressive strength of $Al_2O_3$ nano-particles added with A356 alloy increase at 0.5 wt% and then decrease. The compressive strength increase is due to significant grain refinement, presence of reasonably distribution of hard particle. Dislocation generation due to thermal mismatch, load transfer from matrix to reinforcement and orowan strengthening. The compressive yield strength is as cast increased by addition of 0.5 wt% $Al_2O_3$ .In the T6 condition the compressive strength decrease at 0.5 wt% of $Al_2O_3$ nano-particles

due to less resistance offered by the hard ceramic particles against the applied load during compressive strength. And also $Al_2O_3$ nano-particles not dissolve in solid solution at 400 $^0$C to 540 $^0$C in solutionizing heat treatment.

**4.** The nano-dispersed alloy containing 0, 0.5 and 1 wt% of $Al_2O_3$ nano-particles exhibited the highest strength of double shear in both as-cast and T6 heat treated conditions. The nano-dispersed alloy containing 0, 0.5 and 1 wt% of Al2O3 nano-particles exhibited the highest strength of double shear in as-cast. Beyont1 wt% of Al2O3 nano-particles exhibited the lower strength of double shear. Due to the fracture of the reinforcing particles, partial de-bonding of particle, that leads to lower strength.

## References


[1] M.R. Ghomashchi and A. Vikhrov, "Squeeze casting: an overview," Journal of material Processing Technology., No101,1-9(2000).

[2] EL.Mahallawi ,H.Abdelkader and L.Yousef, "Influence of $Al_2O_3$ nano-dispersions on microstructure features and mechanical properties of cast and T6 heat-treated Al Si hypoeutectic Alloys," material science & engineering A., No 06-061(2012).

[3] S.A. Sajjadi, M. Torabi Parizi, H.R. Ezatpour and A. Sedghi, "Fabrication of A356 composite reinforced with micro and nano $Al_2O_3$ particles by a developed compocasting method and study of its properties," Journal of alloys and compounds., No511, 226-231(2012).

[4] D.Emadi , L.V. Whiting, and M.Sahoo, "optimal heat treatment of A356.2 alloy," light metals 2003,TMS, 2003.

[5] S.A. Sajjadi, H.R. Ezatpour and H. Beygi, "Microstructure and mechanical properties of Al –$Al_2O_3$ micro and nano composites fabricated by stir casting," Journal of materials science and engineering A., No 528, 8765-8771(2011).

[6] Peng Ji-hua ,Tang Xiao-long and He Jian-ting, "Effect of heat treatment on microstructure and tensile properties of A356 alloys ,"Trans.Nonferrous Met.Soc.China., No 21, 1950-1956(2011).

[7] A. Ansary Yar , M. Montazerian , H. Abdizadeh and H.R. Baharvandi , "Microstructure and mechanical properties of aluminum alloy matrix composite reinforced with nano-particle MgO," Journal of alloys and compounds.,No484, 400-404(2009).

[8] Shuangjie Chu and Renjie Wu, "The structure and bending properties of squeeze cast A356 aluminium alloy reinforced with alumina particles," Composites Science and technology.,No 59, 157-162(1999).

[9] H.R Ezatpour and H.Beygi, "microstructure and mechanical properties os Al- $Al_2O_3$ micro and nano composites fabricated by a novel stir casting route," 2$^{nd}$ conference on application of nanotechnology in science, engineering and medicine, mashhad-iran., 16-17 may (2011).

[10] El-Sayed Youssef El-Kady, Tamer Samir Mahmoud, and Mohamed Abdel-Aziz Sayed, "Elevated Temperatures Tensile Characteristics of Cast A356/$Al_2O_3$ Nano composites Fabricated Using a Combination of Rheo-casting and Squeeze Casting Techniques," Journal of material science and applications., 2, 390-398(2011).

[11] Mohsen Hajizamani and Hamidreza Baharvandi, "fabrication and studying the mechanical properties of A356 alloy reinforced with $Al_2O_3$ -10% vol.$ZrO_2$ nano particles through stir casting," advance in materials physics and chemistry., No1,26-30(2011).

[12] S.A. Sajjadi, H. R. Ezatpour and H. Beygi, "Comparison of microstructure and mechanical properties of A356 aluminum alloy/ $Al_2O_3$ composites fabricated by stir and compo-casting processes ," Journal of Materials and Design., No 34 ,106-111(2012).